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REGRESSION MODELS TO PREDICT LEVELS OF AOX GENERATION

Project 3826

Report 1

A Progress Report

to the

NATIONAL COUNCIL OF THE PAPER INDUSTRY FOR AIR AND STREAM IMPROVEMENT

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TABLE OF CONTENTS

SUMMARY	1
OBJECTIVE	2
BACKGROUND	2
AOX Prediction Equations	2
APPROACH	4
RESULTS AND DISCUSSION	5
DATABASE ASSEMBLY	5
AOX FORMED IN CHLORINATION AND CAUSTIC EXTRACTION STAGES	
(CE AOX)	7
Atomic Chlorine as the Only Independent Variable	7
Individual Data Sets	7
Grouped Data Sets	7
Nonlinearity	9
Separation of Effects of Cl_2 and ClO_2 in the	
First Stage	11
Search for a Global Model; Effects of Substitution and	
Unbleached Kappa Number	13
Effects of Filtrate Preparation	13
AOX GENERATION FOR ATOMIC CHLORINE LESS THAN 3% IN THE	
FIRST STAGE	13
AOX GENERATION IN LATER BLEACHING STAGES	14
AOX GENERATION IN THE BLEACHING OF HARDWOOD PULPS	14
CONCLUSIONS	18
ACKNOWLEDGEMENT	19
LABORATORY DATA SETS	20
OTHER REFERENCES	22

SUMMARY

Data on AOX generation in laboratory bleaching were collected from 23 sources. A database was constructed to include the bleaching conditions and levels of AOX measured. Initial statistical analysis of the database showed a good correlation between AOX and % atomic chlorine applied. Atomic chlorine is defined as:

$$\% \text{Atomic chlorine} = \% \text{ on pulp as Cl}_2 + 0.526(\% \text{ on pulp as ClO}_2)$$

The % atomic chlorine applied ranged from near zero to 10% on a dry pulp basis. Data on "C"E AOX generation in the chlorination stage and first caustic extraction stage, when expressed as a function of atomic chlorine, were found to fall into three homogeneous groups and one heterogeneous group. Limited data on AOX generation in hardwood bleaching suggest that less is formed per unit of atomic chlorine consumed than in softwood bleaching.

Regression analysis was done on a subset of the database where the atomic chlorine in the first stage was 3% or less. For softwood pulps, the overall predictive equation for AOX generation was:

Softwood Pulps (A1T less than or equal to 3%)

$$\text{AOX} = 1.04(\text{A1T}) - 0.156(\text{A1T}^2) + 0.0132(\text{A1T})(\text{KAPPANO}) + 0.204(\text{A2})$$

This analysis included linear and quadratic terms for atomic chlorine as well as interaction terms between atomic chlorine and Kappa no., percent substitution, and presence of oxygen delignification. For softwood pulps, this analysis showed both linear and quadratic terms for atomic chlorine (A1T) and the A1T*Kappa No. interaction to be significant. AOX generation in the later bleaching stages was found to correlate directly with atomic chlorine applied in these stages (A2).

OBJECTIVE

The objective of a study currently underway at the Institute of Paper Science and Technology is to construct mathematical models capable of accurately predicting effects of pulping and bleaching process changes on the levels of adsorbable organic halides (AOX) and chlorophenols generated in the bleach plant.

The objective of Phase 1 of the study, now complete, was to correlate AOX data generated under controlled conditions in the laboratory.

BACKGROUND

Previous work on prediction of AOX levels from consumptions of individual bleaching chemicals is briefly reviewed here.

AOX Prediction Equations

An early effort to develop quantitative predictions of chlorinated organics levels was that of Germgård and Larsson (1983), who proposed the following equation for calculation of total organically bound chlorine (TOCl) in bleach plant effluents:

$$TOCl = k(C + 0.5H + 0.2D)$$

where C, H, and D are consumptions of chlorine, hypochlorite, and chlorine dioxide, respectively, each being expressed as active chlorine per unit weight of pulp, and k is a dimensionless constant in the range 0.07 to 0.11. TOCl is determined by a different procedure from AOX and tends to be 0-30% lower. The coefficients of H and D in the above equation effectively convert them to a chlorine atom basis; for example, 0.2D is the number of kilograms of chlorine atoms contained in the chlorine dioxide consumed.

Assuming a constant proportionality between TOCl and AOX thus allows us to write the following equation:

$$AOX = k(Cl)$$

where AOX is the amount of AOX generated in kg per ton of pulp; k is a dimensionless constant; and Cl is the total weight of chlorine atoms consumed per unit weight of pulp, regardless of the type of chemical that contains them. This quantity will be referred to here as "atomic chlorine." Axegård (1988) found values of k between

0.08 and 0.15 for a full bleach sequence, and Earl and Reeve (18) found a value of 0.10 for the (DC)E and CE partial sequences when Cl was expressed as kg per ton of dry pulp.

Lindström and Nordén (1990) proposed an equation containing separate terms for the chlorine and chlorine dioxide in the first bleaching stage:

$$AOX = k_1 (Cl)_{CD-C} + k_2 (Cl)_{CD-D}$$

where $(Cl)_{CD-C}$ and $(Cl)_{CD-D}$ are the respective contributions of chlorine and chlorine dioxide to the atomic chlorine in the "chlorination" stage.

Furthermore, they neglected AOX formation in the D_1 and D_2 stages, an assumption reflecting the fact that the first bleaching stage more efficiently converts chlorine atoms to AOX than brightening (D_1 and D_2) stages. The AOX formed in the latter stages is, however, probably too large to be neglected, suggesting that the addition of another term would improve this equation.

To predict the AOX output of a variety of technology trains, McDonough (1992) used the following equation:

$$AOX = 0.09 (Cl)_C + 0.01 (Cl)_D$$

where $(Cl)_C$ and $(Cl)_D$ refer to the atomic chlorine in the first bleaching stage and in the brightening stages, respectively, each being expressed in kg/ton. This equation accounts for the attenuated AOX formation in the brightening stages, but, like those of Germgård, Axegård, and Reeve, it does not differentiate between the chlorine and chlorine dioxide in the first stage.

Data reported by Liebergott (15) suggest the existence of another effect which is a candidate for incorporation into the model. He showed that the extent of conversion of Cl to AOX (the quantity represented by k in the above equations) decreases as the level of substitution of chlorine dioxide for chlorine is increased above 70%. McCubbin et al. (1992) incorporated this effect into the equation they used, as follows:

$$AOX = 0.08 [1.7 - (\%Substitution/100)] (C + 0.5H + 0.2D) (1 - e_B)$$

where e_B is the AOX removal efficiency of the biological treatment system. This equation recognizes the substitution effect, but neglects the differences between chlorine dioxide and chlorine in

the first stage and between the first stage and the brightening stages.

In summary, a variety of equations for predicting AOX have been proposed, but none takes into account all of the effects that have been shown to be important. Another shortcoming of the existing equations is that each has been developed from a restricted set of data and none has exploited the commonality between data sets to achieve a greater degree of generality. The work reported here addresses both of these deficiencies.

APPROACH

The basic approach consisted of the following steps:

1. Survey the literature to identify potentially useful laboratory "C"E and DED AOX data sets.
2. Identify candidate models for CE AOX (e.g., CE AOX vs. atomic chlorine [AlT], CE AOX vs. chlorine in first stage and atomic chlorine in chlorine dioxide in first stage, etc.).
3. Fit each eligible candidate model to each of several representative data sets and compare models for intralab application.
4. Collect all data sets into a single spreadsheet-type database.
5. Assign dummy variables to distinguish data sets and use multiple regression analysis to divide them into homogeneous groups with respect to the simple atomic chlorine correlation for CE AOX.
6. Separate the hardwood pulp data from that for softwoods.
7. Derive the best available correlation of CE AOX with atomic chlorine (AlT) for each homogeneous group.
8. Derive the best available correlation with AlT and AlT^2 for each group.
9. Discard any data sets found to be atypical in step 3 above and seek a universal correlation for CE AOX by obtaining and comparing regressions on subsets of the following independent variables: total atomic chlorine in stage 1, (AlT), AlT^2 , $AlT \cdot \text{Substitution}$, $AlT \cdot \text{Kappa}$, and $AlT \cdot \text{Oxygen}$.

10. For data sets that qualify, do regressions of total AOX minus "C"E AOX on atomic chlorine in chlorine dioxide in stages applied after the caustic extraction stage.
11. Combine the results of steps 9 and 10 to obtain the best available "global" model for AOX.
12. Prepare separate database to include all points where AlT is less than or equal to 3%. Do regressions on this data set as in Steps 7 and 8 above.

RESULTS AND DISCUSSION

DATABASE ASSEMBLY

A search of the literature uncovered 23 separate sets of data that appeared suitable for the present study. All were for kraft pulp, and nearly all were for softwoods. Most, but not all, were found in published articles; a few were from unpublished reports and meeting presentations. Figure 1 provides an overall view of the softwood data points in the form of a plot of AOX formed in the first two stages against the total charge of chlorine atoms. The R^2 for a linear regression of these data points is 0.933.

The data had been generated by bleaching both unbleached and oxygen predelignified pulps over a wide range of levels of active chlorine charge and chlorine dioxide substitution. Most of the data related to AOX generation in the first ("chlorination") and second (caustic extraction) stages, corresponding either to combined filtrates or individual filtrates from these two stages.

Some sources reported levels of AOX generation in chlorine dioxide stages following the caustic extraction stage; these were used for separate assessment of rates of generation in the two types of stages.

The sources from which the data sets were obtained are listed at the end of this report in arbitrary order.

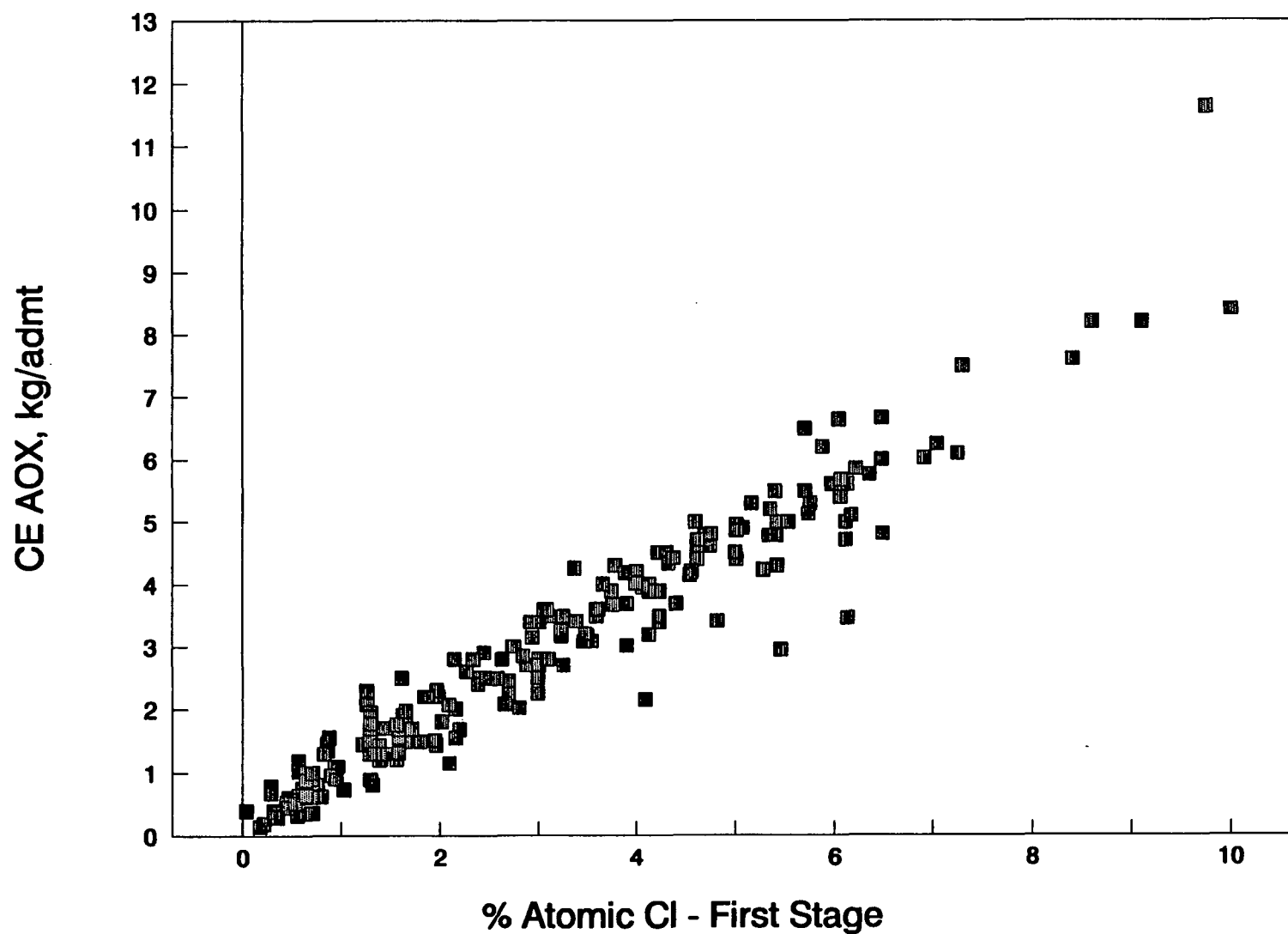


Figure 1 CE AOX as a function of atomic chlorine in the first stage for all references.

AOX FORMED IN CHLORINATION AND CAUSTIC EXTRACTION STAGES (CE AOX)**Atomic Chlorine as the Only Independent Variable**

As discussed in the Background section, numerous authors have developed correlations between AOX formed in the chlorination and caustic extraction stages (CE AOX) and a quantity proportional to the number of chlorine atoms applied per unit of dry pulp mass. This quantity, calculated as the sum of the chlorine charge and the chlorine dioxide charge (expressed as actual chemical) multiplied by its fractional chlorine content (0.526), will be referred to here as the total first stage atomic chlorine charge and denoted by the symbol AlT. Linear correlations of AOX and AlT are often observed.

Individual Data Sets Eligible data sets were individually used to develop correlations of this type, with the results shown in Table 1. It is apparent that the linear correlation is a reasonably good predictor of AOX. On the other hand, there are significant differences between the various correlations with regard to both slope and intercept. In addition, the scatter around the lines, as measured by the standard error, varies considerably from one case to the next.

Grouped Data Sets An effort was made to divide the total database into a small number of homogeneous groups, within each of which no significant differences exist. This was done by defining dummy variables, one for each data set. The dummy variable for a given data set was assigned a value of one for all points within that data set and was assigned a value of zero for all points not in that data set. These variables were then included as independent variables, together with AlT, in a multiple regression having CE AOX as the dependent variable. The significance of the coefficient of each dummy variable was then tested; significant dummy variables signaled a departure of the general level of the AOX data in that group from the level characteristic of those data sets whose dummy variables did not have significant coefficients (the "common group"). Additionally, the possibility that a given group had a different slope from that of the common group was investigated by testing the significance of the coefficient of the cross-product of that group's dummy variable with AlT.

By proceeding in this fashion, the database was divided into three homogeneous groups (Groups A, B, and C) and one heterogeneous group (Group D). Each of the homogeneous groups can be characterized by a single correlation with AlT, but the correlations are significantly different from group to group. The heterogeneous group consists of data sets having unique correlations, all differing significantly from one another. The group to which each

data set belongs is indicated in the last column of Table 1. The regression equations for each group are given in Table 2.

**TABLE 1: CORRELATIONS OF CE AOX WITH ATOMIC CHLORINE
FOR INDIVIDUAL DATA SETS
SOFTWOOD ONLY**

Ref.	Intercept	Slope	R ²	Standard Error	Number of Points	Group
3	0.675	0.602	0.946	0.11	5	A
8	0.691	0.693	0.923	0.35	11	A
10	0.235	0.825	0.984	0.22	7	A
21	0.168	0.825	0.863	0.13	6	A
22	0.268	0.793	0.833	0.20	9	A
4	0.373	0.872	0.995	0.12	7	B
13	1.390	0.250	0.085	0.18	20	B
18	0.060	0.951	0.936	0.54	15	B
17	-0.377	0.994	0.997	0.08	7	C
20	0.163	0.657	0.969	0.09	7	C
2	0.287	1.442	0.975	0.11	13	D
5	0.491	0.772	0.908	0.39	20	D
11 ^a	-0.020	1.167	0.997	0.06	24	D
12	0.100	0.529	0.992	0.12	5	D
14	0.795	0.802	0.943	0.30	26	D
16	0.860	0.748	0.869	0.34	21	D
23	-0.312	1.12	0.984	0.32	31	D

Note: ^aRegression results biased since the data points from this source were values calculated from a fitted equation rather than raw data.

Figure 2 graphically portrays the relationships between CE AOX and atomic chlorine for the homogeneous and heterogeneous groups.

**TABLE 2: CORRELATIONS OF CE AOX WITH ATOMIC CHLORINE
FOR HOMOGENEOUS GROUPS
SOFTWOOD ONLY**

Group	Intercept	Slope	R ²	Standard Error	Number of Points
A	0.260	0.762	0.968	0.25	40
B	0.491	0.864	0.972	0.38	45
C	-0.165	0.909	0.974	0.18	14

Nonlinearity The possibility of significant curvature in the correlations with A1T was investigated by testing the significance of the coefficient of an A1T² term in regression analyses of several of the individual data sets. Table 3 shows the results, which provide some evidence for negative curvature.

**TABLE 3: QUADRATIC REGRESSIONS OF CE AOX ON A1T
FOR SELECTED INDIVIDUAL DATA SETS
SOFTWOOD ONLY**

Ref.	Intercept	Linear Term	Quadratic Term, b ₂	Signif ^a . of b ₂	R ²	Standard Error
18 ^b	-1.15	1.48	-0.0495	ns	0.945	0.52
10	-0.08	1.12	-0.0439	*	0.993	0.17
11 ^c	-0.08	1.27	-0.0279	*	0.997	0.06
14	-0.02	1.25	-0.0545	*	0.954	0.28
8	-0.38	1.31	-0.0760	ns	0.942	0.32

Notes:

^ans = not significant; * = significant at 5% level of α .

^bData points at kappa factor greater than 0.3 were discarded before conducting the regression; leaving them in causes the quadratic term to become significant.

^cRegression results biased since the data points from this source were values calculated from a regression equation rather than raw data.

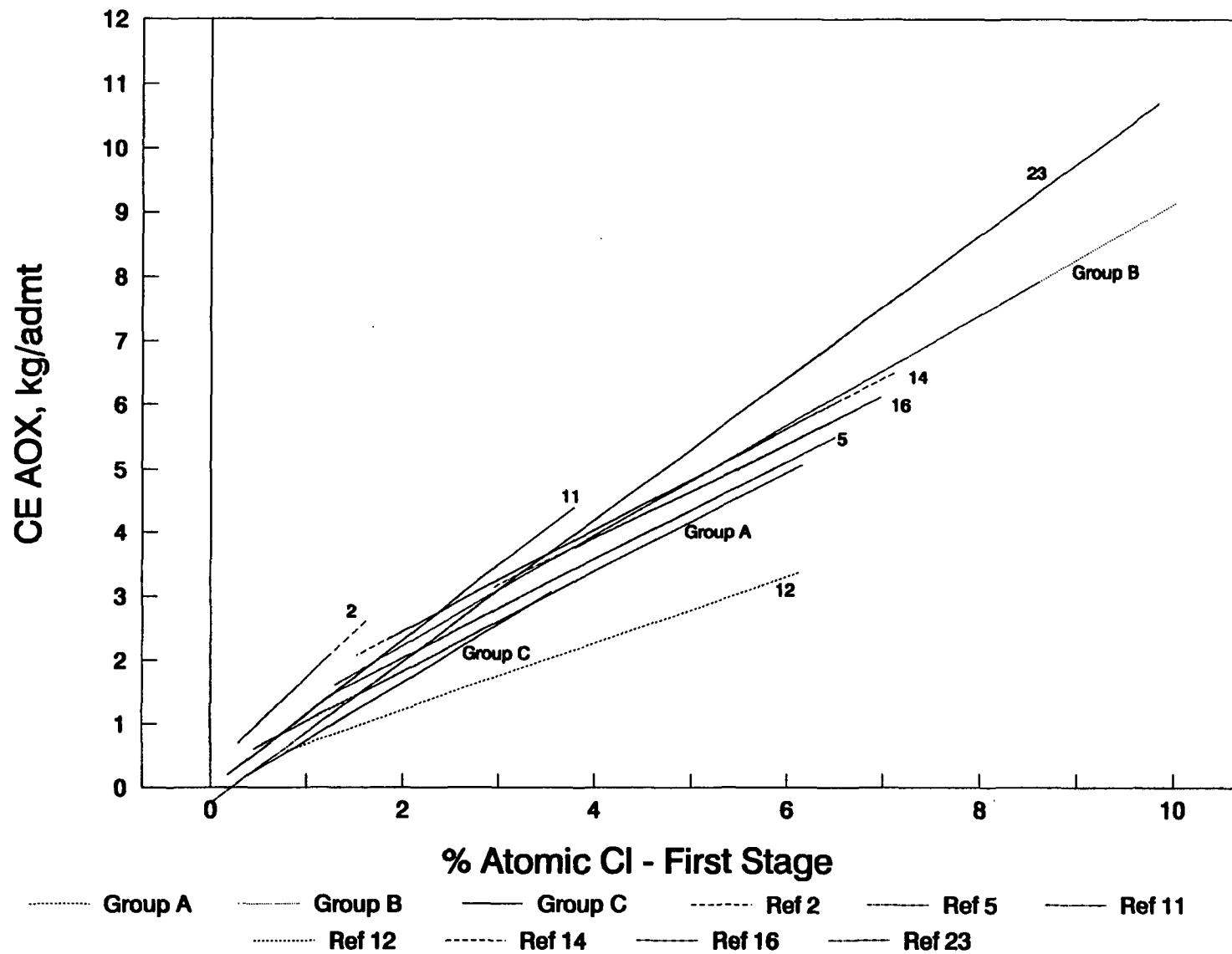


Figure 2 Regression lines of data groups for CE AOX vs. atomic chlorine.

The results of similar regressions on the homogeneous groups are shown in Table 4. Again, there is some evidence of negative curvature. Group C, which exhibits positive curvature, has relatively few data points and may be unduly influenced by several points obtained at 100% substitution under conditions of relatively high breakthrough.

**TABLE 4: QUADRATIC REGRESSIONS OF CE AOX ON A1T
FOR HOMOGENEOUS GROUPS
SOFTWOOD ONLY**

Group	Inter- cept	Linear Term	Quadratic Term, b_2	Signif ^a . of b_2	R^2	Standard Error
A	0.067	0.971	-0.0297	*	0.972	0.24
B	0.305	1.000	-0.0145	ns	0.974	0.37
C	0.164	0.393	0.128	*	0.987	0.14

Note: 'ns = not significant; * = significant at 5% level of α .

Separation of Effects of Cl_2 and ClO_2 in the First Stage

Lindström and Nordén in 1990 proposed a model that provided for different rates of generation of AOX from chlorine and chlorine dioxide in the first stage. To investigate this, such a model was fitted to an arbitrary selection of individual data sets (Table 5) and the homogeneous groups (Table 6). In these regressions, A1C denotes the atomic chlorine corresponding to the molecular chlorine in the first stage and A1D denotes the atomic chlorine contained in the chlorine dioxide added to the first stage.

Comparison of Table 5 with Table 1 reveals little to be gained by introducing the additional complication of separate coefficients for A1C and A1D, except in the case of reference 14, where the new model was a significant improvement. This was due to an apparently much greater rate of generation of AOX from chlorine dioxide than from chlorine. Comparison of Table 6 with Table 2 shows no corresponding advantage of the new model.

**TABLE 5: REGRESSIONS OF CE AOX ON A1C AND A1D
FOR SELECTED INDIVIDUAL DATA SETS
SOFTWOOD ONLY**

Ref.	Inter- cept	Coeff. of A1C, b_c	Sign. ^a of b_c	Coeff. of A1D, b_n	Sign. ^a of b_n	R ²	Std. Error
18 ^b	-0.238	0.984	*	1.643	*	0.945	0.52
10	0.062	0.832	*	1.239	*	0.990	0.19
11 ^c	-0.008	1.166	*	1.131	*	0.997	0.07
14	-1.227	1.101	*	2.404	*	0.977	0.20
8	2.668	0.386	ns	-1.319	*	0.937	0.33

Notes:

*ns = not significant; * = significant at 5% level of α .^bData points at kappa factor greater than 0.3 were discarded before conducting the regression; leaving them in causes the quadratic term to become significant.^cRegression results biased since the data points from this source were values calculated from a regression equation rather than raw data.

**TABLE 6: REGRESSIONS OF CE AOX ON A1C AND A1D
FOR HOMOGENEOUS GROUPS
SOFTWOOD ONLY**

Group	Inter- cept	Coeff. of A1C, b_c	Sign. ^a of b_c	Coeff. of A1D, b_n	Sign. ^a of b_n	R ²	Std. Error
A	0.025	1.180	*	0.809	*	0.970	0.246
B	0.293	0.890	*	1.020	*	0.973	0.384
C	0.006	0.859	*	0.623	*	0.978	0.173

Note: ** = significant at 5% level of α .

Search for a Global Model; Effects of Substitution and Unbleached Kappa Number

Further possibilities are available for an improved AOX model. Liebergott (15) and McCubbin (1992) proposed a model incorporating a lower rate of conversion of Cl to AOX at high substitution levels. The current authors proposed a dependence on unbleached kappa number. To test these ideas, in the hope of finding a more universally applicable model, all of the softwood data were combined and subjected to stepwise multiple regression analysis, allowing the program to choose from among the following variables: $A1T$, $A1T^2$, $A1T \cdot \text{SUBSTITUTION}$, $A1T \cdot \text{KAPPANO}$, and $A1T \cdot \text{O2DELIG}$. An overall equation was found to represent the range of data points in the references up to 10% $A1T$. However, it was concluded that this was not completely relevant as this range does not represent current or anticipated industry practice. Further analysis was restricted to data points where the atomic chlorine in the first stage was equal to or less than 3%.

Effects of Filtrate Preparation

The literature references were investigated with regard to the experimental methods used, particularly with regard to whether the filtrates from individual stages were measured for AOX separately or were combined before measuring. In most of the references, these details were not discussed and inferences could only be made about the filtrate handling from the way the data were reported. From the few references (1, 12, 21) where the filtrates were separately measured for AOX, there were no obvious conclusions that could be drawn regarding the level of AOX measurements compared to the cases where filtrates were mixed before measuring.

AOX GENERATION FOR ATOMIC CHLORINE LESS THAN 3% IN THE FIRST STAGE

A separate data set was established for all data points where the atomic chlorine charge in the first stage was less than 3%. There were 127 points included in this set. The initial analysis showed that one data set (Ref. 12) was substantially different from the rest, so it was not included in this regression. The results of the regression analysis using $A1T$, $A1T \cdot \text{KAPPANO}$, $A1T^2$, $A1T \cdot \text{SUBSTITUTION}$, and $A1T \cdot \text{O2DELIG}$ showed that $A1T$, $A1T^2$, and $A1T \cdot \text{KAPPANO}$ were significant. The equation found was:

$$\text{CE AOX} = 1.04(A1T) - 0.156(A1T^2) + 0.0132(A1T)(\text{KAPPANO})$$

where CE AOX = kg/admt; $A1T$, and $A2$ = %atomic chlorine on o.d. pulp

The standard error for this equation is $s = 0.307$. The initial regression for this database showed that the intercept was a non-significant term. The regression was consequently run by forcing

the line through the origin to produce the above equation. The R^2 value is not reported since it is artificially inflated by forcing the equation through the origin. The inclusion of the $A1T^2$ term in this regression indicates significant curvature over this range. The data points and regression lines for selected Kappa nos. of 32 and 18 are shown graphically in Figure 3.

AOX GENERATION IN LATER BLEACHING STAGES

It was previously recognized that atomic chlorine added to the pulp as chlorine dioxide after the caustic extraction stage is less efficiently converted to AOX than that added before the extraction stage. To quantify this, those data sets having data on both CE AOX and total AOX were used to seek a relationship between AOX generation and atomic chlorine in later bleaching stages.

Regression of the difference between total AOX and CE AOX on atomic chlorine applied after the E stage showed a significant relationship with the atomic chlorine applied in the later bleaching stages (A2). The equation that resulted was:

$$\text{TOTAL} - \text{CE AOX} = 0.204(A2)$$

where $s = 0.13$ for 45 data points. The intercept was found to be non-significant so the equation was forced through the origin. As before, the R^2 value is not reported since it is artificially inflated by forcing the equation through the origin. The data and regression line are plotted in Figure 4. This equation may be added to the CE AOX equation on page 14 to provide a predictive equation for AOX from the entire bleach sequence for the case where the atomic chlorine applied in the first stage is less than 3%:

$$\text{AOX} = 1.04(A1T) - 0.156(A1T^2) + 0.0132(A1T)(KAPPANO) + 0.204(A2)$$

Data points from the data sets for total bleach sequence AOX along with regression lines for two Kappa nos. of 32 and 18 at each of three A2 levels are shown graphically in Figure 5.

AOX GENERATION IN THE BLEACHING OF HARDWOOD PULPS

Regression analysis led to the conclusion that smaller amounts of AOX are generated in the first two stages of hardwood pulp bleaching than in the corresponding stages of softwood bleaching. On the other hand, the very small number (9) of hardwood data points available made it inadvisable to either report a separate equation or to place a high degree of confidence in this result.

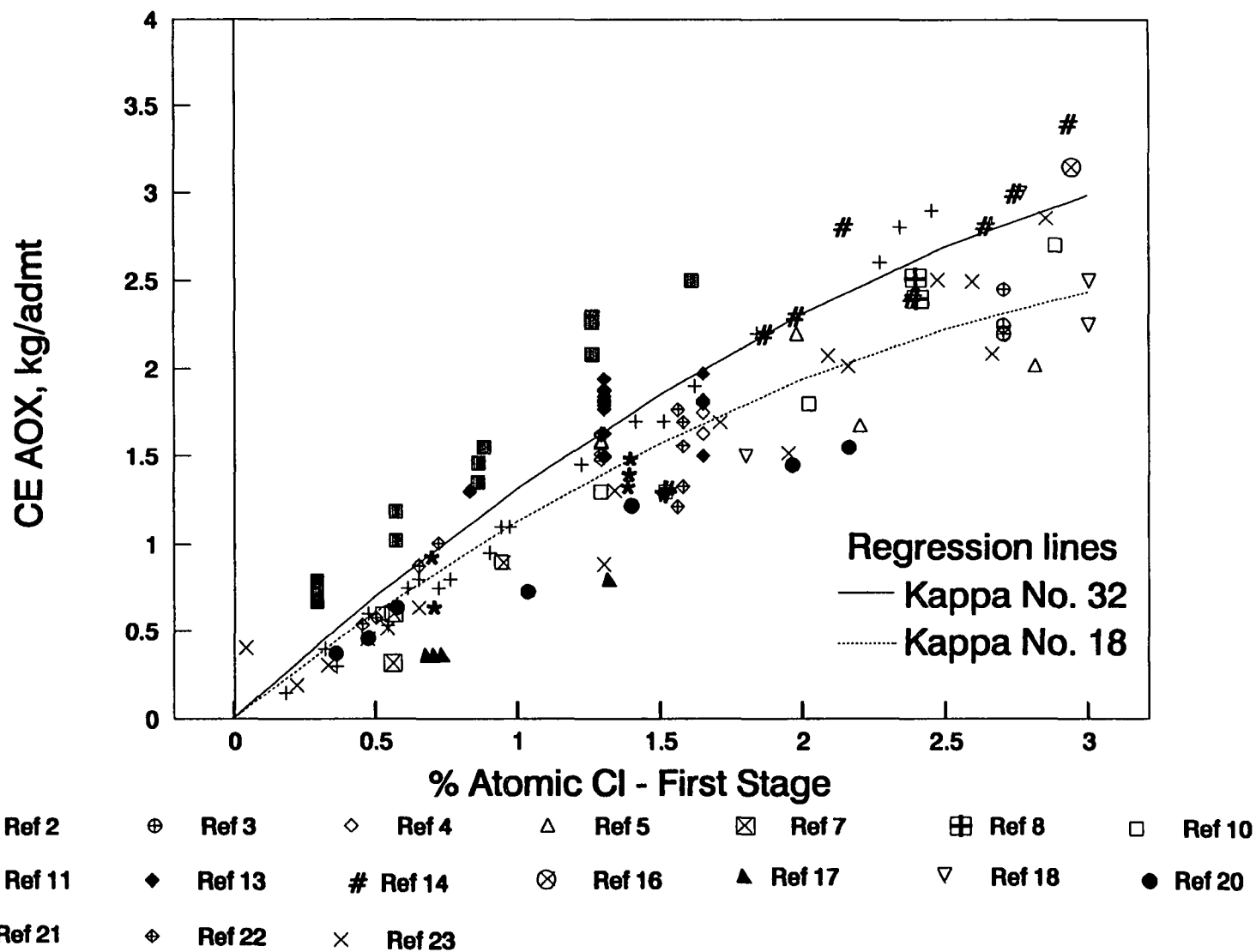


Figure 3 CE AOX as a function of atomic chlorine in the first stage.

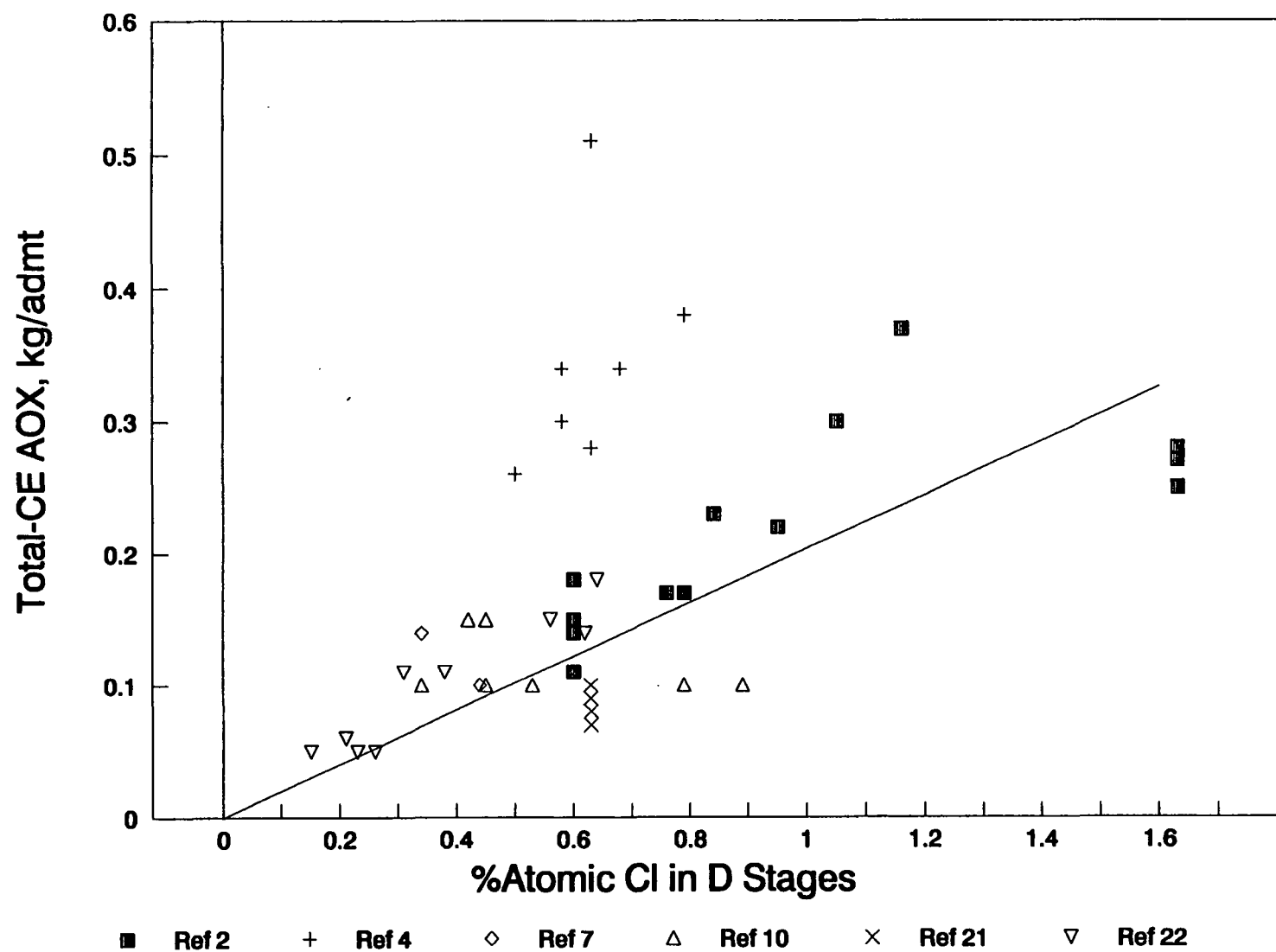


Figure 4 **Difference of total and CE AOX as a function of atomic chlorine**

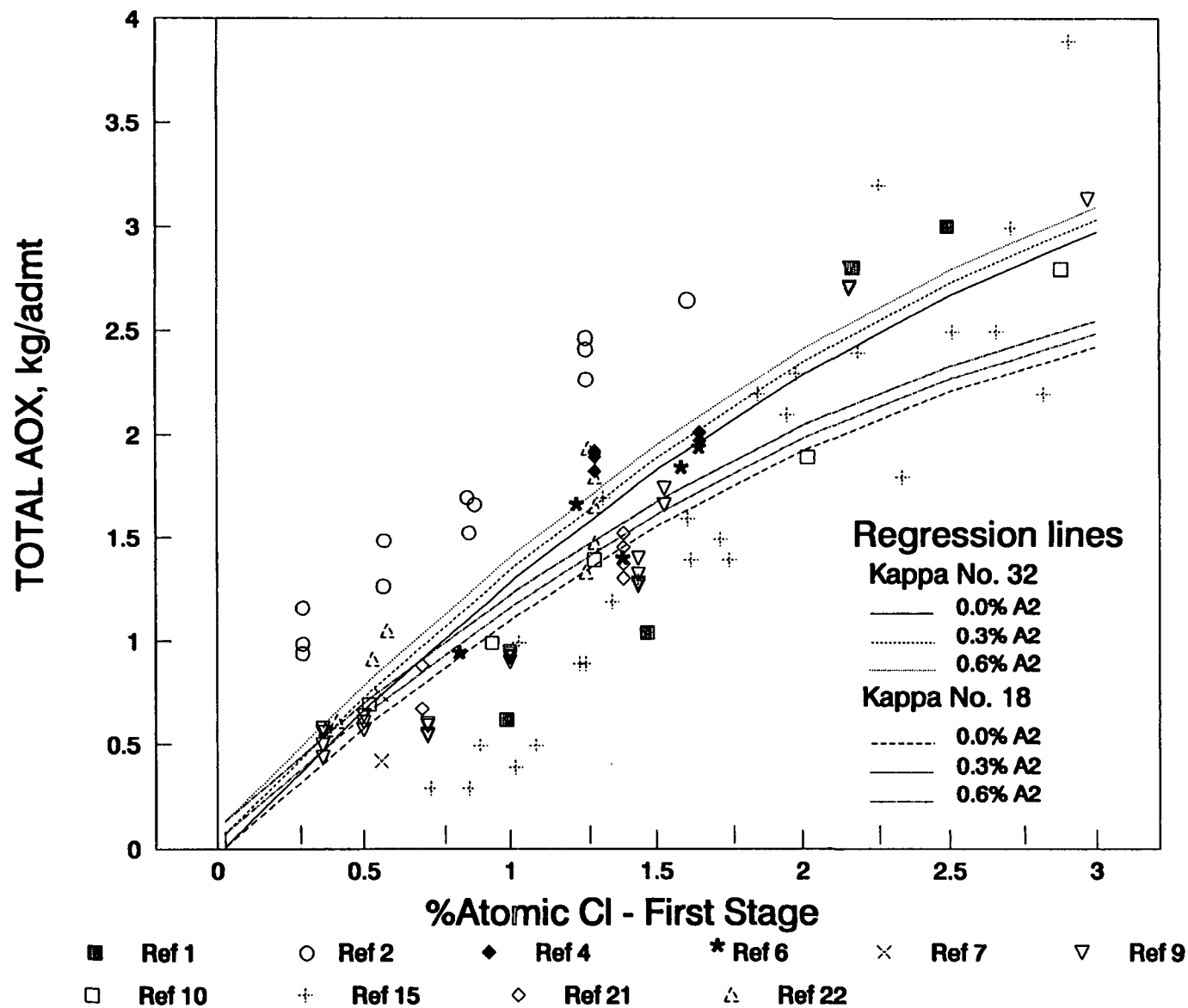


Figure 5 Total AOX as a function of first stage atomic chlorine .

CONCLUSIONS

A statistical analysis of 23 published sets of data on the effects of laboratory bleaching conditions on AOX generation showed the following:

1. For softwood pulp bleaching, a linear correlation was found for AOX generation in the first two bleaching stages ("C"E) and the total atomic chlorine charge in the first stage. There was no statistically significant difference in AOX generating propensity between atomic chlorine from Cl_2 or ClO_2 in the first ("C") stage. The level of AOX generation was somewhat higher with increasing unbleached Kappa no.
2. Differences between data sets could not be readily explained by ascertainable experimental differences. Experimental differences which could potentially account for such differences as mixing of filtrates prior to AOX determination were not detailed in many of the references. For several references where the "C" and E stage filtrates were kept separate for AOX determination, there was no obvious difference in AOX level compared to references where they were mixed before testing.
3. The contribution to total bleach plant AOX from the later stages is also dependent on the atomic chlorine used in these stages. The rate of generation is much less than that from the first two stages.
4. For data where the atomic chlorine applied in the first stage (A1T) is less than 3%, the regression equation for AOX generation was found to be:

Softwood (A1T less than 3%)
$$\text{AOX} = 1.04(\text{A1T}) - 0.156(\text{A1T})^2 + 0.0132(\text{A1T})(\text{KAPPANO}) + 0.204(\text{A2})$$

where A1T = % atomic chlorine applied in the first stage, and
A2 = % atomic chlorine applied in later bleaching stages.

5. Only a limited number of data points were available for hardwood. The AOX generation from hardwood was indicated to be somewhat less than that from softwood at the same level of atomic chlorine consumption.

ACKNOWLEDGEMENT

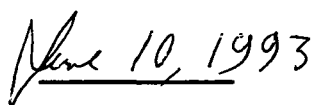
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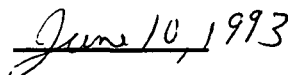
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Date

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